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AUGUST 1940.

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Volume 5.

Volume 5 of the series of *Annual Reviews of Petroleum Technology* is published at the beginning of September 1940. It covers developments in the industry during 1939 and the early part of 1940. The published price is 11*s*., and members of the Institute and subscribers to the *Journal* may purchase one copy at 5*s*. 6*d*. (post free to United Kingdom and overseas). A prospectus of the book is included with the present number of the *Journal*.

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The object of this information is to assist the Council in grading candidates according to the class of membership.

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HOLE, Herbert Wray. General Manager, New Brunswick Gas and Oilfields, Ltd., Moncton, New Brunswick. (*C. A. P. Southwell; A. E. Dunstan.*) (*Trans. from A.M.*)

JOHNSTON, Douglas Gordon, Engineer, Texas Oil Co., London. (*N. L. Anfilogoff; W. A. Webster.*)

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Vol. 26. No. 202. August 1940.

THE MANUFACTURE AND TESTING OF ROOFING-FELTS AND DAMP-COURSES.*

By D. M. Wilson, M.C., B.Sc., A.I.C.

Introduction.

Little is known about the early use of roofing-felt; but it appears that about 150 years ago, Dr. Faxe, a Swedish naval adviser, nailed paper to a boarded roof, coated it with hot wood-tar, and finished it with coloured mineral earths.

In 1844 ships' sheathing-paper was dipped into a melted mixture of pine-tar and pitch, the excess squeezed out, and the surface sprinkled with sand. At a later date the paper in the form of rolls was run through saturators containing heated tar.

The roofing-felt and the asphalt industries seem to have developed simultaneously, and in Europe both received a stimulus from their use in the waterproofing of magazines and fortifications. Owing to faults in the early days, the use of mastic asphalt in Germany was generally restricted to the covering of horizontal roofs and those with a small slope, so the arched surfaces of bridges and tunnels were proofed with laminated felt imported from England. This appears to have been the material known as "sheathing" felt, made from flax waste and manufactured by David Anderson in 1849. The name was derived from its use as an underlay to the copper plates of ships. Manufacture in Germany began about 1857. Although in France and England mastic asphalt was at first the more favoured waterproofing medium, in Germany the use of felt rapidly outstripped that of asphalt, because tar for impregnation was available in most cities, whereas the asphalt rock had to be carried considerable distances.

It was not until many years after the invention of sheathing felt that it became popular as a roof-covering. When used for this purpose it was coated, directly after laying, with a mixture of pitch and tar, and then sprinkled with sand; with periodical dressings of tar it provided an economical and satisfactory form of roof-covering. Considerable quantities were supplied during the Crimean War, and the War Office continued to use this type of felt up to the end of 1914, when supplies of flax and jute became scarce. Its durability was remarkable, and some of the felt supplied during the Crimean War was still in service at Shorncliffe camp in 1918. Thus a felt originally used for damp-proofing found a use for roofing.

Mention has been made of the early use of paper coated with tar following the crude method of coating brown paper *in situ* with tar; developments along this line followed, chiefly in America. A soft sheet of fibre made on a paper-making machine was treated with tar and sold in rolls. This material had the advantage that it did not require any coating treatment when first laid on the roof. At a later date asphaltic bitumen

* Paper presented to a meeting of the Asphaltic Bitumen Group of the Institute of Petroleum held in London, on Tuesday, 16th April, 1940.

came into use. Although asphaltic bitumen was introduced into Germany from America soon after 1890, tar maintained its dominant position in the roofing-felt industry there until a few years ago. In this country stearine, water-gas, oil-gas, and coal-tar pitches were used for many years before they were replaced by asphaltic bitumen.

Many types of roofing-felt are manufactured to-day, and will be considered under the names given in the British Standard Classification for Bituminous Roofing Felts, no. 747. Similarly, damp-courses will be referred to under the headings given in Part 2, B.S.S. 743. Actually there is really no hard-and-fast division; roofing-felts are quite frequently used for damp-courses, and damp-coursing felts have been used for roofing. Both can be made on the same machines and from the same materials.

Bitumen Felts.

Roofing-felts of this type all consist of a saturated fibre base which may be given a coating either on one side only, or on both.

Fibre Base.

The fibre base, sometimes known as the "rag felt," or the "dry felt," is prepared on a paper-making machine from selected rags containing a large proportion of wool, cotton, and linen, together with jute and Manila fibres. Sometimes a proportion of wood pulp is introduced. The base is made in different thicknesses, ranging usually from about 20 to 80 mils, according to the thickness of the bitumen felt it is desired to make. The composition of the base is of considerable importance; the larger the proportion of wool, cotton, and linen, the better will be the felt. Wool fibres are the most desirable, and cotton and linen come next in durability, whilst jute fibres deteriorate more rapidly. Fillers should not be employed, as their presence would affect the porosity of the felt.

The amount of saturant that the base will hold in its fibres and voids depends largely on its composition and on the skill of the maker, and can be expressed as the weight of bitumen carried by 100 grams of the felt base. In practice the "percentage saturation" may vary from 100 to 180. The waterproofing qualities of the finished felt depend on the presence of a high percentage of bitumen, so the higher the percentage saturation of the base, the better.

Although the fibre base should be very porous, it must nevertheless have sufficient tensile strength to be handled on the saturating- and coating-machines without breaking; the strength is considerably increased by saturation, as shown in Table I.

Asbestos felt may also be used as the fibre base for roofing and damp-coursing felts, and is made in a similar way to rag felt. It usually contains about 85 per cent. of asbestos fibre, and cow-hair is often added to increase the strength and the percentage saturation. Typical properties in the unsaturated and saturated condition are also indicated in Table I.

The asbestos in this example is the thinner by 10 mils, but although its tensile strength is lower in the unsaturated condition, it is considerably higher when saturated. It is saturated and coated in exactly the same manner and on the same machine as the fibre-base felts.

TABLE I.

Type of base.	Thickness of felt, mils.	Saturation, %.	Tensile strength at 20° C. — lbs. per 1 inch wide; test-piece 2 inches wide, 7 inches between grips, load applied at rate of 65 lb. per minute.			
			Dry-fibre base.		Saturated-fibre base.	
			Cut lengthwise.	crosswise.	lengthwise.	crosswise.
Rag felt .	50	139	15	13	37	29
Asbestos felt.	40	87	6	4	51	41

Saturant.

The saturant for the fibre base is usually an asphaltic bitumen of the straight-run type having a penetration at 25° C. of between 100 and 200, and it calls for no particular comment. The harder the saturant the greater will be the tensile strength of the saturated fibre; if it is too hard, however, the pliability of the finished felt will be affected.

Coating.

A coating of asphaltic bitumen is applied to one or both sides of the saturated fibre to act as a seal and fill any voids in the fibre that may remain after saturation. The coating is usually a blown bitumen, and some of the roofing-felt manufacturers prepare their own blown grades, in which the properties are adjusted according to the country to which the felt is being supplied.

A high melting point is necessary to withstand exposure to high sun temperatures, but at the same time the felt must remain pliable over a wide range of temperature. If the coating is too hard it is liable to crack while being laid, and moisture may gain admittance to the fibre base. A blown bitumen has a higher melting point for a given penetration than straight-run material, and is more pliable over a range of temperature. This fact is indicated in Table II, where 10 represents a high and 1 a low degree of pliability, the tests being made on strips of bitumen $\frac{1}{10}$-in. thick.

TABLE II.

Type of bitumen.	Melting point (R. & B.). ° C.	Penetration at 25° C.	Penetration index.	Pliability. Temp. of test, ° C.									
				25.	20.	15.	10.	5.	4.	3.	2.	1.	0.
Blown X . .	85·5	34	+ 4	10	10	10	10	10	10	10	10	10	10
Blown Y . .	82·0	18	+ 2	10	10	10	10	6	4	4	4	4	4
Straight-run .	61·1	24	− 0·5	10	10	10	10	1	1	1	1	1	1

A column has been included to show the penetration index * of the bitumens. This method of indicating the temperature susceptibility is most useful, and the table also indicates that for practically the same

* Pfeiffer and Van Doormal, *J. Instn Petrol. Tech.*, 1936, **22**, 414.

melting point the bitumen with a high penetration index is more pliable over a range of temperature than the one having a lower index.

Blowing of Bitumen.

The properties of a blown bitumen can be varied according to the method of blowing employed and the "stock" from which the bitumen is blown.

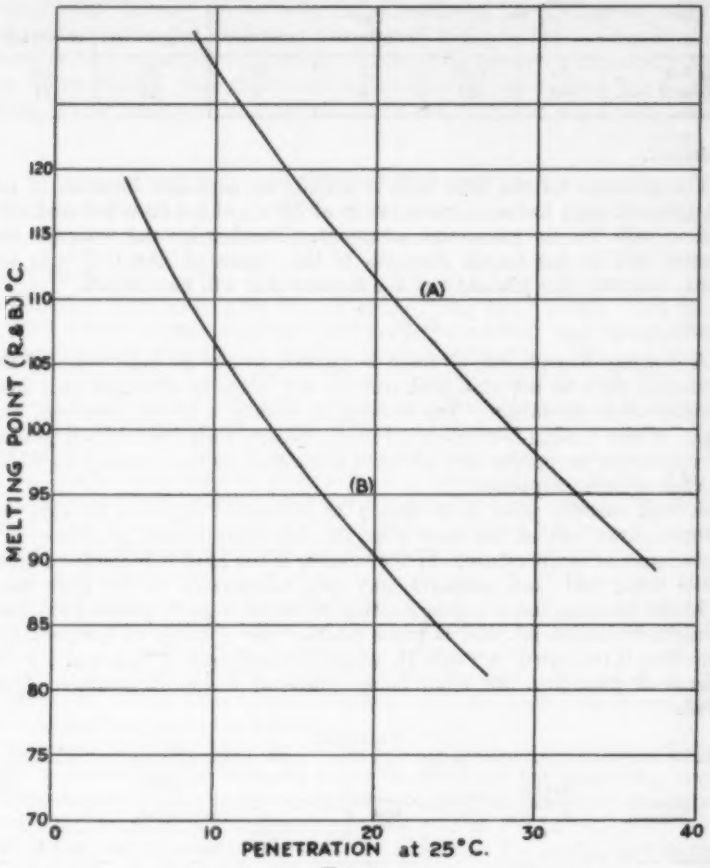

FIG. 1.

CURVES SHOWING MELTING POINT/PENETRATION RELATIONSHIP OF BITUMEN BLOWN (A) UNDER PRESSURE AND (B) BY NORMAL METHOD.

In order to obtain both a high melting point and a high penetration, the blowing stock must be in the nature of a very heavy asphaltic oil, and the process should be conducted so that the maximum oxidizing effect is obtained with the minimum loss of light oils. One method of obtaining such conditions is to blow the bitumen in a partly closed con-

tainer under pressure. The air is introduced at a pressure of 50–60 lb. per square inch, the outlet to the container is partly closed, and the volume of air is adjusted so that the pressure in the vessel is 20–30 lb. per square inch. Fig. 1 shows the effect on the penetration/melting-point curve when the same material—a very heavy fuel oil—is blown under pressure and when no pressure is applied, all other conditions being kept constant. The application of pressure leads to the production of a bitumen with a higher penetration index, as the loss of distillate is reduced. When heavier material, such as 500 penetration bitumen, is used as the blowing stock, a blown bitumen results which has a lower penetration index.

A bitumen with a high penetration index may also be obtained by fluxing a bitumen blown to a high melting point—say, 135° C.—with some of the distillate produced in the blowing process, or with a flux oil. One disadvantage, however, of some of the high-penetration-index blown bitumens is that they may suffer a considerable loss when heated to, say, 200° C. Table III shows the loss on heating undergone by two blown bitumens of practically the same melting point, but different penetration indices. The bitumen with the high penetration index shows a much higher loss on heating than the one with the lower index.

TABLE III.

Blown bitumen.	Melting point (R. & B.). °C.	Penetration at 25° C.	Penetration index.	Loss on heating 5 hrs. at 200° C. %.
Type 1 . .	80·5	35	+ 3·5	2·3
Type 2 . .	80·0	23	+ 2·5	0·5

It is often necessary to compromise and use a bitumen with a lower penetration rather than run the risk of a high loss in subsequent heating processes. "Sweating" of the light oils from the finished felt may occur with a bitumen of high penetration index and tend to discolour the talc or other dressing. Judgment has to be employed, therefore, in choosing bitumens of suitable properties. In a general way it may be said that for this country the most suitable coating is a blown bitumen having a melting point of about 85° C. with a penetration at 25° C. between 20 and 30. Most manufacturers have drawn up their own formulæ after many years of experience, various additions being made to the blown bitumen to improve its weathering properties and pliability.

Dusting and Finishing Materials.

The dusting-powder is talc, mica, sand, or other suitable powder, which is spread on the surface of the coated felt as it passes through the machine. The object of the dusting is to prevent the felt from sticking to the rollers of the machine and when in the rolled condition. It also increases the thermal reflectivity when the felt is laid on a roof. Care must be taken not to apply too heavy a dressing, as it may present difficulties when the felt is being laid. The dusting compound should be removed as far as possible when two surfaces are being stuck together, as on a built-up

roof, or it will interfere with the adhesion of the bitumen used to cement the layers.

Various-coloured grits are also applied to the finished surface of some types of felt instead of a dusting-powder, and are lightly pressed into position by rollers while the bitumen coating is still warm. These grits protect the bitumen from the ageing effect of sunlight, particularly if they are light in colour, and at the same time improve the appearance of a roof. The grits may either be coloured naturally, or in some of the brighter colours may consist of a slate or granite coloured artificially.

Manufacture of Bitumen Felts.

A modern machine is shown diagrammatically in Fig. 2. The fibre base is received in rolls, and is unwound on to a "Looper" (*A*), which

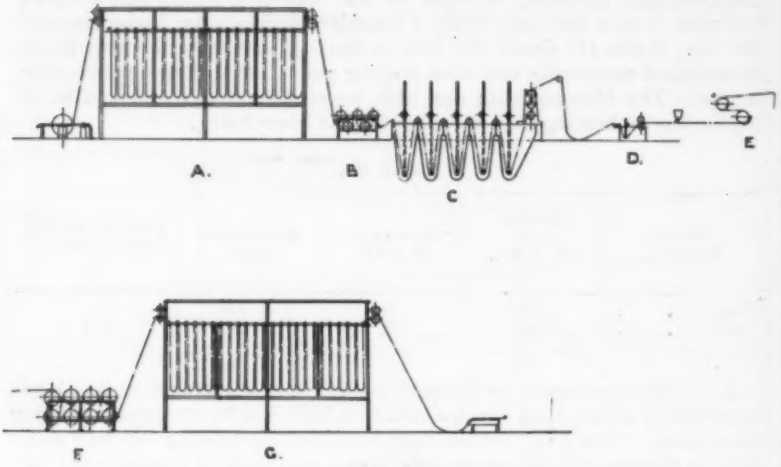

FIG. 2.

DIAGRAM OF FELT SATURATING AND COATING MACHINE.

builds up a reserve so that the machine need not be stopped to splice on a new roll of fibre. The fibre passes over drying-cylinders (*B*) and then through the saturator (*C*). It passes to the coating-rolls (*D*) on to the first cooling-rollers (*E*), where the dusting material is applied, and then to further cooling-rollers (*F*) and another looper (*G*), where it is finally cooled and stored before reeling. The tanks holding the saturant and coating may be jacketed and heated by the circulation of hot oil from a central heater such as that indicated in Fig. 3, which is operated by gas burners (Fig. 4). The temperature of the oil on leaving the heater is maintained automatically at 295° C. The jackets of the bitumen supply-pipes from the storage-tanks to the machines are also heated by hot oil from the circulator.

The machine (Fig. 5) will make all the types of felt detailed in B.S.S. 747, viz.—

1 *A*. *Impregnated Bitumen Felts.*—These, more often referred to as

'saturated" felts, merely consist of the fibre base impregnated with bitumen at a temperature of 150–200° C. Any moisture remaining in the fibre is expelled and evaporated from the surface of the saturant. Any excess of saturant on the surface of the fibre is removed by passing the felt through heated rollers, and it is then cooled and rolled.

1 *B. Sanded Bitumen Felts.*—A somewhat harder saturant is employed than in the previous type, and some is allowed to remain on the surface. Sand is used as the finishing material. Although still employed abroad, the method is practically obsolete in this country.

1 *C. Self-finished Bitumen Felts.*—The saturated fibre is passed into the hot coating mixture and then through a pair of steam-heated rollers, where the coating is spread evenly over the surface of the saturated fibre and bonds with the bitumen saturant. The thickness of the coating is regulated by the distance the two rollers are set apart. The felt may be given a "veined" finish by allowing the rollers to rotate, or may be smooth finished if the top roller is kept stationary while the felt is pulled through; the underside is smoothed by applying a scraper-blade to the felt after passing over the bottom roller. After dusting, the felt is cooled on rotating cylinders (Fig. 6) through which water is circulated.

1 *D. Mineralized Bitumen Felt.*—In this class the top side of the felt is finished with coloured grit. A 2-inch margin of the felt may be left free from granules so that a lap can be made during laying. The manufacturing process is largely the same as for the self-finished felts, except that the felt passes through additional press-rolls to ensure that the grit is well bedded. For satisfactory results attention must be paid to the grading of the grit; it should all be retained on an 18-mesh B.S.I. sieve and completely pass the 8-mesh.

1 *E. Reinforced Bitumen Felts.*—The strength of a self-finished felt is considerably increased by adding a layer of hessian. The fibre base and the hessian are passed through the coating process together and become firmly bonded. The felt is used for many purposes, particularly as a sarking under slates.

Impregnated Flax and Hair Felts.

Although this type of felt is not now so extensively employed for roofing purposes, on account of the necessity for periodical dressing, it is still used as an underlay for slates, tiles, and asphalt, and acts as a thermal insulator. It also insulates the asphalt from any movements of the underlying structure.

The base consists of cow-hair or long-fibred flax and jute waste, or mixtures of all these materials, which are carded and thoroughly mixed in a machine similar to that used in the preparation of cotton for spinning; all the dust is extracted, and the fibres remain. The fibres are passed through the preparing-machines, in which they are separated by intimate contact with a rapidly rotating striking-cylinder, the surface of which is fitted at about 2-inch centres with stout iron pins. The fibres are delivered to a rotating perforated cylinder under suction, which extracts the dust and delivers the fibres as a soft "batt" to a flat table,

where it is carried forward to the actual felt-machine for impregnation. It then passes through two rollers, which remove the surplus saturant. Whilst still warm the saturated felt is treated with fine dust derived from flax waste (termed "shives," and consisting mainly of the straw fibre of flax), to prevent the laps from adhering in the rolls. The felt then passes through another series of rollers to compress it further and smooth the surface, and is finally rolled up.

The felt was originally made to suit the width of the Muntz metal plates used for wooden ships. The plates were 32 inches by 20 inches and the felt was cut into pieces 32 inches by 40 inches, each sheet thus being sufficient for laying under two of the metal plates. The felt is still manufactured in a 32-inch width, as are all felts in Part 2 of B.S.S. 747. These are:—

2 *A*. *Impregnated Flax Felt, Black.*—The base consists of flax and jute and the saturant is a fluxed coal-tar pitch.

2 *B*. *Impregnated Flax Felt, Brown.*—Felts in this class are generally described as inodorous, and are intended for use where the smell of coal-tar pitch is not desired. The base is the same as in class 2 *A*, but they are impregnated with mixtures containing wood tars and pitches, together with resins.

Similar brown and black felts are also made in which animal hair forms the batt instead of flax and jute, and are classified under the headings, 2 *C* and 2 *D*.

Damp-coursing Felts.

From the earliest days of the industry, bituminous felts found a use in the damp-coursing of buildings and bridges. At first they were made from flax felt, strips being cut across the width of the roll, giving a length of 32 inches. These strips, after dipping into a pan of hot tar, were placed on a pile of sand; additional sand was sprinkled on top, and then another length was placed on top of the first. When the workman had prepared his strips in this manner, he proceeded to lay them on the brick-work. About 30 years ago the modern type came into use. It consists of a fairly thick bitumen coating on a hessian or fibre base. Sheet lead is also introduced to give added protection against the percolation of water in wet situations.

The components are the same as for roofing-felts, and the same type of bitumen is used as the saturant for the hessian or fibre base. Blown bitumens generally form a considerable part of the coating, and the penetration index may with advantage be higher than in the case of roofing-felts, in order to maintain the pliability of the somewhat thicker coating. This is usually given greater stability by the addition of a filler, such as slate dust, limestone, asbestos, or asbestine. The melting point of the coating can be adjusted by the addition of fluxing oils to suit widely differing conditions, and satisfactory results have been obtained both in the tropics and in the coldest Northern climates.

Attention must be paid to the pliability of the coating to avoid cracking when a roll is laid in cold weather, although it is true that with suitable coatings the cracks are self-healing when under load in a wall. The

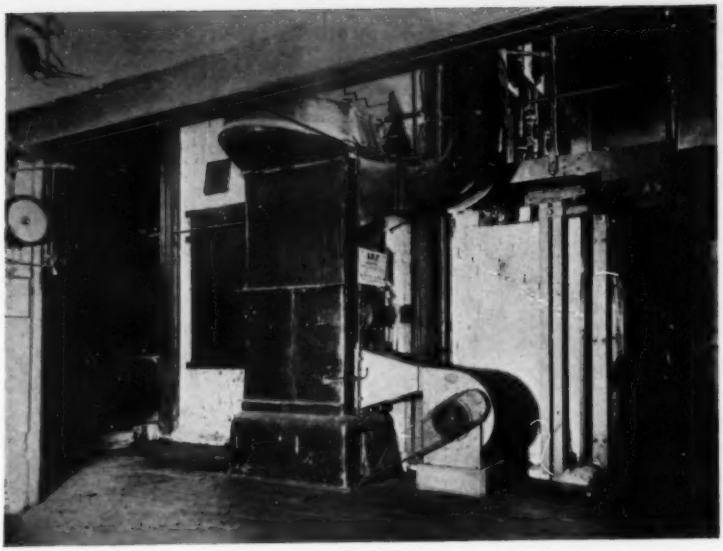

FIG. 3.
CENTRAL OIL-HEATING INSTALLATION.

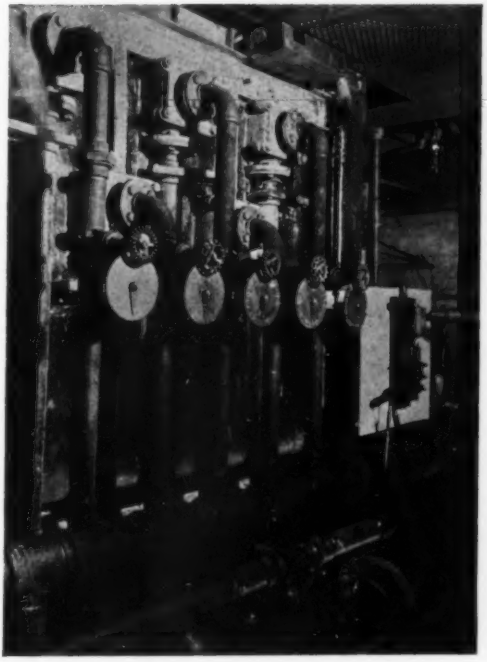

FIG. 4.
GAS BURNERS OF OIL HEATER.

[*To face p.* 384.

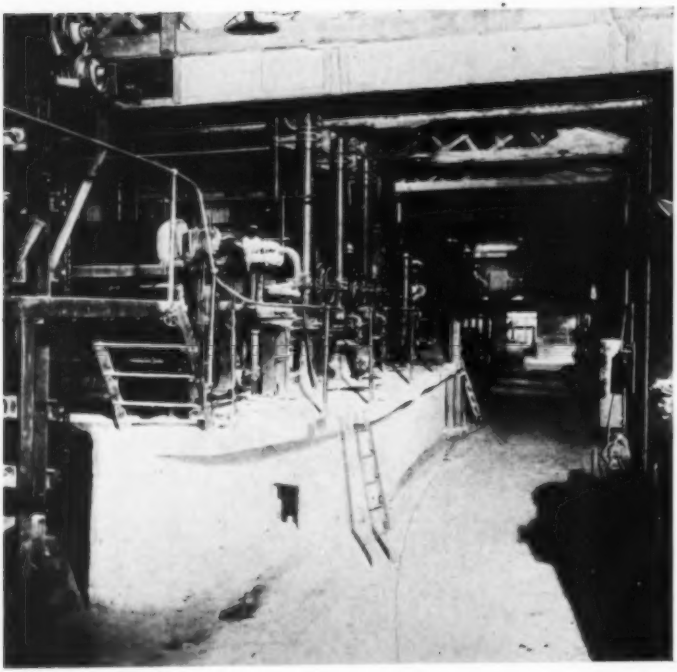

FIG. 5.

BITUMEN FELT MACHINE.

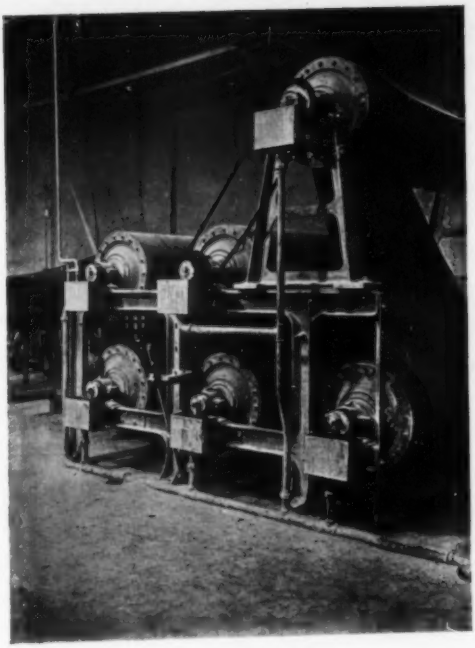

FIG. 6.

COOLING ROLLS.

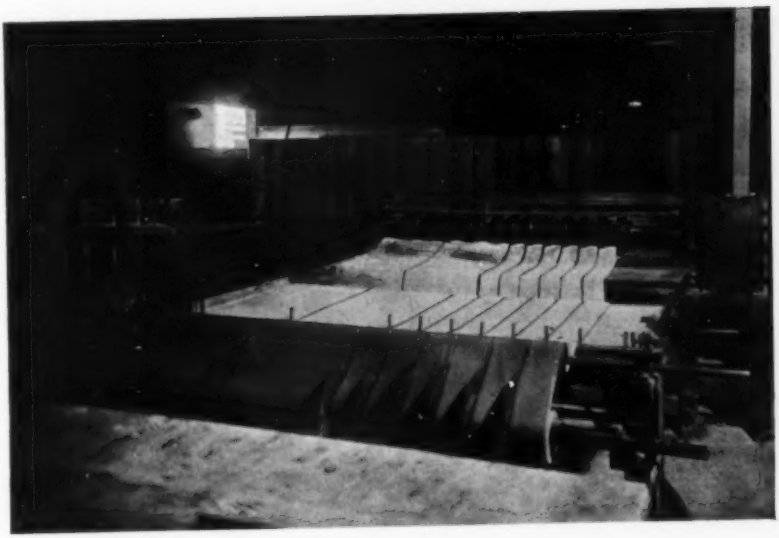

FIG. 7.

CUTTING MACHINE FOR DAMPCOURSING.

coating must also be sufficiently stable, or there would be a risk of its partial extrusion when laid.

Damp-course felts are generally made in 3 or 6 feet widths, which are cut to the width required by a machine of the type shown in Fig. 7 and are generally sold in rolls 8 yards long.

Bituminous damp-courses are classified in B.S.S. 743 according to the base materials, viz. :—

Hessian base.
Fibre base.
Lead core and hessian base.
Lead core and fibre base.

Many other types are also on the market in which different combinations of the base materials are employed. As in the case of roofing, asbestos felt may replace the fibre base.

Testing of Roofing-Felts and Damp-courses.

A large number of tests can be applied to roofing-felts, but standardization is difficult, as with all products containing bitumen, and it is not easy to eliminate the personal factor. Fortunately the manufacture of roofing-felts is largely in the hands of firms of repute, some of whom have been in the business for the best part of a century, and they can be relied upon to maintain a high standard.

Tests on Raw Materials.

Fibre Base.—This is tested for thickness by a Leunig micrometer gauge, which exerts a uniform pressure at its contact surfaces, for Mullen strength and for its weight per square yard. Several tests have been described for measuring the saturating capacity, but it is far more satisfactory to saturate a length of the fibre on a machine and then extract a known weight of the saturated material to determine its bitumen content.

Bitumen.—Control of the bitumen is both by melting point and penetration, and rapid methods are preferable. For the melting point test a multiple apparatus * has proved valuable, and for the penetration test the method † that enables a figure to be obtained in 30 minutes has now been used for many years.

Pitch and Tar.—The consistency of the grades used for saturating felt can usefully be controlled by the Float test (A.S.T.M. D.139–27) at a temperature of 75° C.

Finished Bitumen Felt.

Pliability.—One of the most important tests that can be applied to a roofing-felt is that for pliability. A method has been standardized by the A.S.T.M. in which the strips of felt, 6 inches × 1 inch, after immersion in water at an appropriate temperature, are immediately bent through

* *Chemistry and Industry*, 1938, **33**, 767. † *Ibid.*, 1932, **10**, 204.

180° at a uniform speed in exactly 2 seconds around a mandrel. The pliability can be expressed by a number, thus :—

1. Cracks entirely through the sheet on the 25-mm. mandrel.
2. Cracks part of the way through the sheet on the 25-mm. mandrel.
3. Cracks on the 25-mm. mandrel.
4. Cracks on the 20-mm. mandrel.
5. Cracks on the 15-mm. mandrel.
6. Cracks on the 10-mm. mandrel.
7. Cracks on the 5-mm. mandrel.
8. Cracks when bent through 180° over a $\frac{1}{16}$-inch (1·588-mm.) mandrel.
9. May be bent through 180° over a $\frac{1}{16}$-inch (1·588-mm.) mandrel in one direction without cracking, but will crack when bent through 360° in the opposite direction.
10. May be bent through 180° over a $\frac{1}{16}$-inch (1·588-mm.) mandrel in one direction, and then through 360° over a $\frac{1}{16}$-inch mandrel in the opposite direction without cracking.

By making the tests at temperatures ranging from 25° C. downwards, a very good "picture" can be obtained of the pliability of the felt. The thickness of the film has a considerable effect on the results, and in Table IV figures are given for layers of bitumen prepared by pouring the melted material on to an iron plate, treated with dextrin solution to prevent sticking, to the approximate thickness required. The thickness is finally adjusted by passing the bitumen sheet when cold through a pair of rollers.

TABLE IV.

Thickness, in.	Pliability. Temp. of test,° C.											
	25.	20.	15.	10.	5.	4.	3.	2.	1.	0.	−2.	−4.
$\frac{1}{32}$	10	10	10	10	10	10	10	10	9	8	6	1
$\frac{1}{16}$	10	10	10	10	10	10	9	9	9	9	5	1
$\frac{1}{8}$	10	10	10	10	5	1	1	1	1	1	1	1

It is obvious, therefore, that the thickness of the coating on a felt must be taken into account when considering the results of pliability tests. More consistent results could no doubt be obtained if the bending of the felt round the mandrel could be done mechanically, but nevertheless in its present form the test provides useful information.

Tensile Tests.—In one Government specification the tensile strength is required to be tested on strips of felt 2 inches wide by 7 inches between grips, cut from the roll both in the direction of its longer length and crosswise, the rate of loading being 65 lb. per minute. The strength is expressed as the load in lb. per inch width required to break the test-piece.

A lever-machine fitted with grips of the type used for textile testing can be employed, the load being applied by either lead shot or water run into a canister hung from the end of the beam.

The test-pieces should be aged for at least one week in the case of newly

manufactured felt. Temperature has a considerable effect on the results, as shown in Table V, and should be quoted with the strength figures. The elongation at the breaking point can also usefully be recorded.

TABLE V.

Tensile strength :— Load in lb. per inch width required to break test-pieces.	Conditioning of test-piece.	
	Exposure for 6 hrs. in atmosphere of 65% R.H. at temp. of 21° C. (70° F.).	Exposure for 6 hrs. in atmosphere of 65% R.H. at temp. of 11° C. (52° F.).
Lengthwise . . .	27	35
Crosswise . . .	20	26

Weathering Tests.—The weathering qualities of a felt may be gauged by making pliability and tensile strength tests on strips that have been exposed in some type of artificial weathering apparatus, such as the Fugitometer.

Surface changes as the felt undergoes ageing may also be recorded by making reflectivity or colour tests on the surface and then repeating the tests at intervals as ageing proceeds. A photo-electric colorimeter may be used for this purpose, a portion of the original surface which has not been exposed being used as the reference standard. In this way changes in the surface characteristics which affect the reflectivity may be recorded as a single number, and the rate of change can give a useful indication of the extent to which the felt will resist the effects of weathering.

Tests on Coating.—Application of the bitumen recovery methods to a felt will only give the properties of the mixture of the coating and the saturant. It is often possible, however, to remove surplus dusting material and then scrape off sufficient of the coating unmixed with saturant for a melting point and penetration test.

Examination of "Dry Felt."—The bitumen may be removed by solvents in a Soxhlet extractor, so that the fibre base may be examined for thickness and weight per square yard. By measuring the thickness of the felt before and after extraction, the thickness of the coating may be determined.

Finished Flax and Hair Felts.—The approximate content of saturant is determined in a Soxhlet extractor using benzene, a thin wad of cotton wool being placed in the side-tube to act as a filter in preventing hairs from passing through. If tar is present a considerable portion of the free carbon comes over with the liquid. After extraction the hair is dried and teased out on a sieve to separate it as far as possible from the free carbon. The weight of the carbon is added to that of the extracted tar, which is obtained after evaporating the benzene and drying to constant weight at 105° C.

The percentage of animal hair may be determined by extracting a weighed amount of the "batt" with 2 per cent. caustic soda. By washing and drying the residue the amount of hair dissolved out can be determined.

Damp-courses.

The same tests are applied as in the case of roofing-felts. In addition, the resistance to the percolation of water may be tested by clamping a test-piece between two flanges, one of them being screwed on to a length of 3-inch pipe supported vertically. The pipe is filled with water and the damp-course can be submitted to the pressure of any desired head of water.

Conclusion.

These briefly are the methods of test in use in the industry at the present time, and suffice for the routine control of production. There are many other possible methods of distinguishing between a good felt and one that is not so good, and they are being explored.

The author's thanks are due to the Directors of Messrs. D. Anderson & Son, Ltd., Stretford, for permission to publish this paper.

DISCUSSION.

The CHAIRMAN (Mr. J. S. JACKSON) commented on the fact that the paper was particularly welcome, as it was probably the first paper on this aspect of the subject given in this country. He was pleased to note that in a modern plant for the manufacture of roofing felts special care was taken to dry the felt before it entered the impregnating bath. If damp felt were used, frothing frequently occurred. It was not always appreciated that quite small quantities of water of the order of 0·02 per cent. would cause asphaltic bitumen to froth, and such quantities could easily be introduced by using damp felt.

He asked the author whether he had experienced any special difficulties due to foaming caused by aeration.

He expressed surprise that manufacturers of roofing felt should still carry out their own "blowing" operations, since the blowing of relatively small quantities of bitumen must be a very inefficient and costly operation.

He referred to the difficulties experienced by the trade in connection with the staining of felts as the result of "sweating," and inquired whether the author had arrived at any satisfactory explanation of this trouble.

He noted that "filled" blown bitumens were used for the coating of damp-courses, and he was interested to know whether the use of lightly blown bitumens filled with a fibrous material had been tried for coating ordinary roofing felts.

In connection with the testing of roofing felts, he suggested that too much importance should not be attached to superficial colour changes resulting from the exposure of felts during the weathering test.

He suggested that a further paper dealing with the use of roofing felt would serve a very useful purpose.

He felt that the use of this excellent material was not widely understood, and its possibilities were certainly not generally appreciated.

Mr. A. V. HUSSEY, as a humble user of roofing felt, suggested that one of the chief weaknesses was its lack of resistance to tearing. Reference had been made to the employment of hessian for the fibre base, and it seemed to him that the incorporation of such a material must inevitably greatly improve the felt, particularly with regard to resistance to tear. In connection with the testing of felts, it occurred to him that the application of the Mullen test, as used for paper, would serve as a valuable index of the quality of a roofing felt. Furthermore, it was a well-known fact that, for example, four or five layers of paper were more resistant to shock and other stresses than the same thickness of paper but not made up of five plys. By analogy, therefore, it would seem that roofing felt made with two or more fibre bases interspersed with bitumen would be stronger than one fibre base covered with increasing thickness of bitumen according to quality and weight.

Reference had been made during the discussion to the possible deterioration and decomposition of vegetable fibre when embedded in bitumen, resulting in damage to the bitumen coating itself. Mr. Hussey was strongly of opinion that no such dissociation or decomposition was likely to occur unless it arose from some interaction between the bitumen or constituents of the bitumen and fibre itself. He quoted cases where cellulosic materials had been removed from ancient buildings after having been embedded in calcareous clay for more than 2000 years without deterioration.

Lastly, in connection with the laying of felt, he did not think it was generally appreciated how important it was that certain preliminary precautions should be taken to avoid the creasing, folding, and bulging which were generally noticeable on roofs covered with roofing felt. An expert linoleum layer would not attempt to fix the linoleum in position until the expiration of two or three or more weeks after unrolling, whereas in the case of roofing felt it was customary to undo the roll and immediately fix on the roof. In such circumstances it was not likely that satisfactory results would be secured while the unevenness of the material permitted of the penetration of weather, draughts, and high winds which ultimately lead to tearing and other damage.

In his reply, the AUTHOR said that no difficulties arose on account of frothing unless the fibre base had not been dried properly. Some manufacturers found it useful to blow their own bitumen, as it enabled them to produce grades which were not normally on the market. "Sweating" often seemed to be due to the use of a bitumen with too high a penetration index for a particular climate: it appeared that there was a tendency for oils to separate from the bitumen, and so cause staining.

Fibrous fillers such as asbestos and asbestine were often used in the coating for roofing felts as well as dampcourses.

Replying to Mr. Hussey, he said that it was only the very light grades of felt that lacked resistance to tearing, and the grades were only intended for use on small temporary buildings. Heavier grades with a comparatively high tensile strength were available for more permanent work.

Felts reinforced with hessian were finding extensive uses in connection with A.R.P. purposes and for the screening of roof lights. If a sheet of this reinforced felt was stuck to the glass, the latter might be shattered by an explosion, but the felt would prevent the pieces from dropping on the workers below. It would also continue to screen the factory lights and enable production to continue.

The Mullen test was used extensively in the testing of the fibre base, but a tensile machine of the type employed for textiles was better for dealing with the finished article.

Roofing felts with more than one layer of fibre had been made for many years, and were preferred in certain countries.

Finally the author agreed that great care should be exercised in the laying of the felt, but that was a subject almost big enough for a separate paper. Most manufacturers recommended that felt should be unrolled, cut to length, and laid out for as long a period as possible before laying on the roof.

STUDIES ON THE SEPARATION OF PARAFFIN WAXES. PART I. PRINCIPLES INVOLVED IN THE FRACTIONAL MELTING OF WAX.*

By M. F. Sawyer, Ph.D., A.M.Inst.Pet., T. G. Hunter, D.Sc., M.Inst.Pet., and A. W. Nash, M.Sc., F.Inst.Pet.

In recent years considerable research has been conducted in the Department of Oil Engineering and Refining, the University of Birmingham, on phase-rule studies of systems important to the petroleum industry. The more recent publications have dealt with dewaxing [1] and double-solvent refining,[2] each subject having been investigated on a quantitative fundamental basis. Further research was commenced with the object of applying established principles to a study of the refining of waxes, the ultimate intention being to apply the results obtained to the sweating and solvent-refining processes employed industrially in the production of refined paraffin wax.

In many investigations—both qualitative and quantitative—on lubricating oil, a physical property such as V.G.C. has been employed successfully to indicate the quality of the oil. V.G.C. cannot be applied to waxes for this purpose, and the selection of a suitable physical property of wax which is characteristic is a matter of considerable difficulty. This paper, which is of an introductory nature, therefore includes a discussion of the choice of a physical property to characterize the wax.

Fundamental Basis of the Investigation—The Nature of Paraffin Wax.

In any study involving phase transformations the initial consideration must be that of the nature of the substance. Attempts have been made in many researches to elucidate the composition of paraffin wax, and there is general support for the view that *n*-paraffins usually predominate. Hydrocarbons of at least one other type may also be present to some extent, probably dependent on the crude from which the wax is derived, but there is no general agreement as to whether these belong to the *iso*-paraffinic and/or naphthenic series. The waxes used in this study have constituents melting over the range 105–142° F. at least, and assuming that the components are *n*-paraffins, it is possible for those members to be present as shown in Table I.

Information concerning the state or nature of paraffin wax may be obtained from a consideration of the behaviour of wax during melting.

A pure substance is characterized by a sharp melting point, this being the temperature at which, on heating, the entire mass melts, or, on cooling, the entire mass solidifies. Paraffin wax, however, does not possess a sharp melting point, and the melting or solidification process takes place over a temperature range. It was first suggested by Gurwitsch [3] that the

* Paper received April 1940.

single *n*-paraffins are partly isomorphous in relation to each other, and that during the solidification of paraffin wax, solid solutions and not chemically pure compounds, separate. In the case of non-polar organic compounds three conditions must normally be satisfied before two substances can form solid solutions :—

(1) the chemical constitutions must be similar;
(2) the crystal structures must be similar;
(3) the molecular volumes must be nearly equal.

TABLE I.

n-Paraffin.		M.W.	M.Pt. (° C.).	M.Pt. (° F.).	B.P. under 15 mm. ° C.
n-Heneicosane . . .	$C_{21}H_{44}$	296·3	40·4	104·7	215
n-Docosane . . .	$C_{22}H_{46}$	310·4	44·5	112·1	224
n-Tricosane . . .	$C_{23}H_{48}$	324·4	47·4	115·3	234
n-Tetracosane . . .	$C_{24}H_{50}$	338·4	51·1	124·0	243
n-Pentacosane . . .	$C_{25}H_{52}$	352·4	53·3	127·9	259
n-Hexacosane . . .	$C_{26}H_{54}$	366·4	56·6	133·9	262
n-Heptacosane . . .	$C_{27}H_{56}$	380·4	59·5	139·1	270
n-Octacosane . . .	$C_{28}H_{58}$	394·4	61·6	142·9	278

Applying these conditions to the *n*-paraffins included in Table I, it is reasonable to suggest, for example, that there is a greater possibility of solid solution formation between *n*-$C_{28}H_{58}$ and *n*-$C_{27}H_{56}$ than between *n*-$C_{28}H_{58}$ and *n*-$C_{21}H_{44}$. Assuming, then, that the paraffin wax consists in the main of members of the *n*-paraffin series, it is probable that on solidification the components would not all simultaneously solidify to form one solid solution, but that different solid solutions would successively separate—the first to be formed consisting of two or more of the higher components.

Except for the indirect evidence of Gault and Boisselet,[4] who stated that the melting point of a mixture of waxes lies between the melting points of the components, the idea of solid solution formation remained unconfirmed by experiment until the work of Myers and Stegeman,[5] who investigated the equilibria existing in mixtures of waxes of different melting points. By repeated carefully controlled fractional distillations under 0·1 mm. pressure of a large quantity of wax, and the ultimate collection of fractions boiling over narrow ranges, waxes of different melting points were obtained. The liquid–solid equilibrium curves were determined for blends of two of these close-cut fractions *A* and *B* over the complete range 0 per cent. *A*–100 per cent. *B*, and the nature of the diagram obtained provides evidence that wax mixtures form a continuous series of solid solutions. This view was confirmed by a similar series of experiments with two other close-cut wax fractions.

More recently, Verdonk [6] has studied the melting curves of pure *n*-paraffins and commercial waxes, and suggests that the behaviour of the commercial waxes may be explained as being that of a mixture of solid solutions of substances which have two enantiotropically related modifications.

The original theoretical ideas of Gurwitsch have therefore received

experimental support, and any attempt to construct an equilibrium diagram applicable to the fractional melting of waxes could reasonably be developed from the basis indicated by the consideration that paraffin wax consists of a series of solid solutions—probably with *n*-paraffins predominating. Various types of solid–liquid equilibrium diagrams are described in the literature, but in view of the observation of Gault and Boisselet, and the findings of Myers and Stegeman, to which reference has been made, any such diagrams applicable to the process of fractional melting of waxes are more likely to resemble those for a continuous series

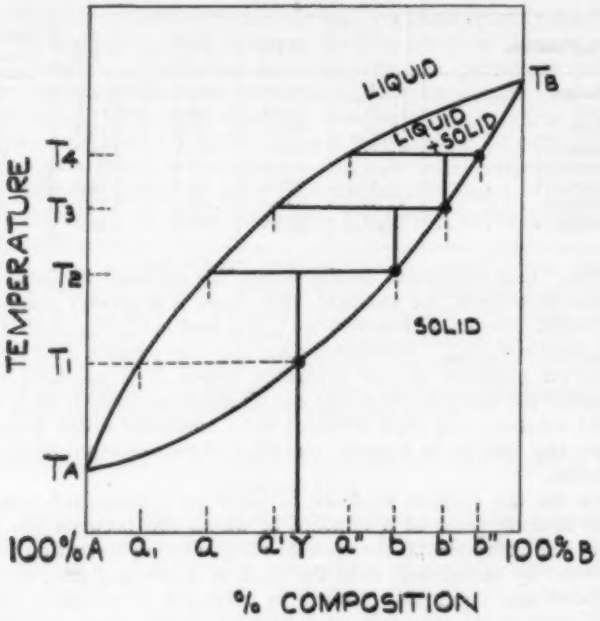

FIG. 1.

of solid solutions rather than those in which solid solutions of minimum or maximum melting point are formed. It is necessary, therefore, to make an elementary and brief study of a typical equilibrium diagram of this nature.

CONTINUOUS SERIES OF SOLID SOLUTIONS—TYPICAL EQUILIBRIUM DIAGRAM.

Fig. 1 is a typical diagram for two substances *A* and *B* which melt respectively at temperatures T_A and T_B. The upper curve is the "liquidus" or equilibrium curve of the liquid solution, and the lower is the "solidus" or equilibrium curve of the solid solution formed by these substances. Any system of composition represented by a point in the

region above the liquidus curve is entirely liquid, and by a point in the region below the solidus curve is entirely solid. Any system of composition represented by a point in the region between the solidus and the liquidus curves is partly liquid and partly solid.

Consider, now, the effect of slowly heating a mixture of composition Y. No phase change occurs until the temperature T_1 is reached, at which stage liquid of composition a_1 appears. At T_2 liquid of composition a co-exists in equilibrium with solid of composition b, and it may readily be shown that the ratio

$$\text{solid} : \text{liquid} = aY : Yb.$$

Let the liquid phase be completely removed at the temperature T_2 and let the remaining solid phase attain equilibrium at temperature T_3, where the liquid phase is of composition a^1 and the solid phase is of composition b^1. Again completely remove the liquid phase and let the remaining solid phase reach equilibrium at T_4, where the compositions of liquid and solid phases are a'' and b'', respectively.

Thus, in this particular three-stage removal of liquid phase the composition of the solid phase changes from Y to b'' and the percentage yield of b'' may be calculated as indicated above.

If, therefore, solid of composition Y is taken at temperature T_1 and the temperature is increased by means of an infinitely large number of infinitesimally small increments, and the resulting liquid phase is removed from the solid after each temperature increase, it follows that the composition of the solid present at any stage will change from Y to 100 per cent. B along the lower curve $T_A T_B$. In this ideal process pure B, the higher-melting component, will therefore ultimately be obtained.

Modified Representation of Equilibrium Diagram for a Continuous Series of Solid Solutions.

Fig. 1 represents the usual and simplest form of the equilibrium diagram for two substances forming a continuous series of solid solutions. It is clear, however, that it may also be represented in the modified form shown in Fig. 2, where composition is characterized by a physical property, in this case freezing point. In this diagram, equilibrium temperature is plotted against freezing point, defined in this case as that temperature at which solid first appears on cooling a liquid containing any proportion of components A and B. It is clear from Fig. 1, that as the property of freezing point does not follow the simple mixture rule, the curve relating freezing point to composition is an essential feature of Fig. 2, in order that computations may be evolved in a manner similar to that indicated for the previous figure.

The liquidus curve is a 45° line, as the freezing point of the liquid phase formed by any mixture is the same as the equilibrium temperature (Fig. 1). The solidus curve in Fig. 2 may easily be obtained by re-plotting the data from Fig. 1.

It follows that if a mixture of the two components, of freezing point T_3, and therefore composition C_3, is allowed to reach equilibrium at a temperature T and then separated completely into the two phases, the liquid phase will be of freezing point T_1 and composition C_1, whilst the

solid phase will be of freezing point T_2 and composition C_2. Also the ratio

$$\frac{\text{Liquid phase}}{\text{Solid phase}} = \frac{C_2 - C_S}{C_S - C_1}$$

If freezing point in any particular case followed the simple mixture law, it is obvious that for purposes of computation the lower curve (Fig. 2) showing freezing point *versus* composition would be unnecessary, as the relative amounts of the liquid and solid phases would be given by $(T_2 - T_S)$ and $(T_S - T_1)$ in Fig. 2, respectively. In such a case, the upper curve of Fig. 1 would have been a straight line.

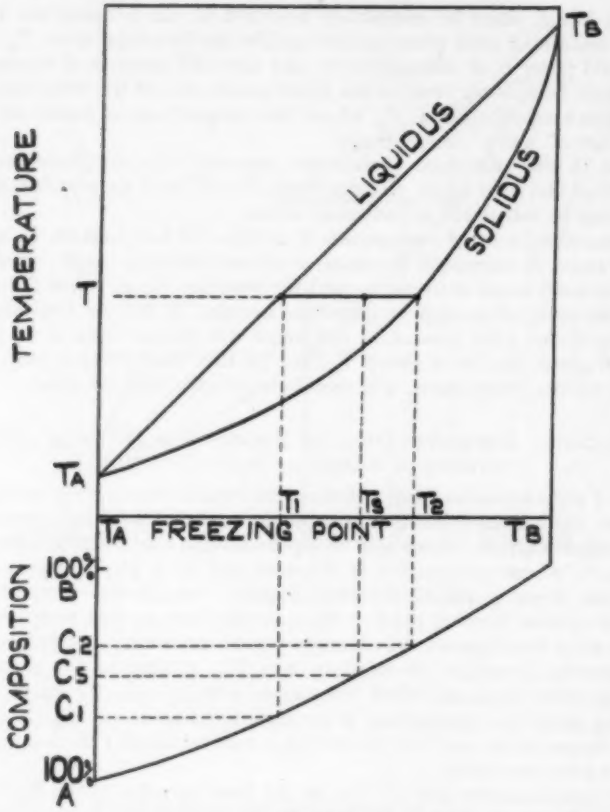

FIG. 2.

DETERMINATION OF EQUILIBRIUM DIAGRAM.

Two general methods may be employed to determine the equilibrium diagram given by substances forming solid solutions:—

(1) *Thermal Analysis.*—A method based on the determination of melting and solidification points for mixtures of known composition:

(*a*) from the study of heating or cooling curves; or (*b*) by direct observation of the melting range; and

(2) *Analysis of Equilibrium Mixtures.*—Allowing a suitable mixture to attain equilibrium at a temperature within the melting range of the components, separating the liquid from the solid phase, and then analysing the two phases.

The first method was used by Myers and Stegeman in the experiments already mentioned, but these workers were dealing with two very close-cut fractions, each melting over a range of not more than 1° C., and which were treated as if they were pure compounds. The actual determination of the melting and solidification points of known mixtures was therefore possible. This method cannot, however, be applied to mixtures of waxes containing unknown quantities of many components, and it is therefore necessary to resort to method (2) above, but even this latter method is not free from experimental difficulty.

Paraffin wax has a high retentive power for hydrocarbon liquids—*e.g.*, oil—and if the last traces of such liquids are to be removed from the wax, special precautions must be taken. It might be anticipated then that solid wax would possess a high retentive power for liquid wax, and that complete separation of the two wax phases formed in the equilibrium experiments would not be easily achieved.

Physical Property for the Characterization of Wax Phases.

The unknown composition of the wax presents an immediate difficulty, and it is therefore necessary to employ some physical property—*e.g.*, molecular weight, melting point, etc.—to characterize the liquid and solid phases formed in the equilibrium experiments in the same way as freezing point has been used to characterize composition in Fig. 2. The essentials of the physical property to be used for this purpose are :—

(1) For maximum convenience it should follow the simple mixture rule—*i.e.*, the physical property of a blend of two waxes should be a linear function of the percentage of the higher (or lower) of the two component waxes, in order that the equilibrium diagram shall apply quantitatively without the use of a secondary curve as in Fig. 2. If a secondary curve has to be employed in the manner illustrated in Fig. 2, then this curve must show the property selected as a function of composition or as a function of another physical property which follows the mixture rule.

(2) It should be capable of accurate determination as the melting range of the wax stock employed is limited, and hence change of physical property with changing composition will be small.

(3) It should be related preferably to the melting point, as this is, from the industrial viewpoint, the most important property of a wax.

As commercially refined paraffin wax contains more than two components, any physical property will be merely an *average* property, and will not, in general, give an indication of the exact composition of the wax. If, for example, three normal paraffins P_1, P_2, P_3, have molecular

weights (in increasing order) of M_1, M_2, M_3, respectively, then there is a very large number of possible blends each having a molecular weight of M, where M is somewhere within the range M_1 to M_3. Since a single physical property is insufficient to define the exact composition of mixtures containing more than two components, any equilibrium diagram for paraffin wax making use of one physical property only will probably have limited applications. This point will be dealt with in detail later.

The physical properties which might possibly be used are as follows :—

(*a*) molecular weight;
(*b*) refractive index;
(*c*) specific gravity;
(*d*) aniline point;
(*e*) melting point.

These properties will now be considered in greater detail.

(a) *Molecular Weight.*—Molecular weight, of course, follows the simple mixture rule, but is difficult to determine accurately in the case of waxes. Using even the ebullioscopic apparatus of Menzies,[7] and extrapolating the molecular weight values to infinite dilution, it is difficult to achieve an accuracy of $\pm$ 2 per cent. consistently.

Considering this limit of accuracy with reference to *n*-docosane and *n*-octacosane—two *n*-paraffins the melting points of which are within the wax range

	M.W.	M.Pt. (° F.).
n-Docosane	310·4	112·1
n-Octacosane	394·4	142·9

it is observed from these values that the melting point is raised by 30·8° F. for an increase of 84 units in molecular weight.

The melting point is therefore affected to the extent of 0·37° F. for each unit increase in molecular weight. Assuming that the molecular weight may be estimated with an accuracy of $\pm$ 1 per cent.—a figure attained only with considerable difficulty in practice—the experimental error on a molecular weight of 300 would be $\pm$ 3 units, equivalent to $\pm$ 1·1° F. With a probable error of such magnitude in the determination, molecular weight is less suitable than melting point for use in the construction of the equilibrium diagram where accuracy to within $\pm$ 0·1° F. is desirable and may be obtained in the melting-point determination. The assumption that the increase in molecular weight per unit increase in melting point for paraffin waxes is approximately equal to the corresponding increase for the two *n*-paraffins considered is justified, since the molecular weight/melting-point curve of the commercial waxes used in this work was found to coincide fairly well with the corresponding curve for *n*-paraffins of molecular weight within the wax range. In this connection it may be noted that Berne-Allen and Work [8] also found close agreement between the experimental values of the molecular weights of the waxes they used and those for the *n*-paraffins of corresponding melting points.

(b) *Refractive Index.*—Refractive index is a property which may be determined rapidly and accurately. It is especially convenient, since only a very small quantity of material is needed for the determination. For waxes of melting point lower than 60° C., the refractive index of the

liquid sample is normally determined at 60° C. For waxes of higher melting point the refractive index may be measured at a temperature 1° C. above the melting point and then reduced to 60° C. by applying a correction of 0·0004°/C. Using an Abbé Refractometer, the refractive index may be estimated to ± 0·0001, and the following are typical values for fractions collected in laboratory "sweatings" of an oil-free wax stock.

TABLE II.

M.Pt. (° F.).	n_D^{60}
112·6	1·4328
119·4	1·4334
127·1	1·4345
137·3	1·4362
142·7	1·4373

These figures indicate that the value of n_D^{60} increases by approximately 0·00015 for each 1° F. increase in melting point, and as n_D^{60} may be estimated to ± 0·0001, the possible error in terms of melting point is more than ± 0·6° F.—an amount which favours the use of melting point rather than refractive index in this connection.

(c) *Specific Gravity.*—Figures given in the literature for the melting point/specific gravity of wax are very variable, but the following data given by Morris and Adkins [9] for American wax may be cited to give an indication of the relation between the two properties :—

TABLE III.

M.Pt. (° F.).	$d_{60°}^{120°}$ F.
103	0·778
109	0·777
115	0·780
118	0·782

It is clear that the specific gravity varies only slightly with melting point, and assuming even that the values for the waxes of 115° and 118° melting point are typical, it is observed that a difference of 3° F. affects the specific gravity by only 0·002. The specific gravity of a wax is not normally required to more than ± 0·001, and as it is doubtful whether the value may be conveniently determined to an accuracy of ± 0·0002, this property is not as accurate as melting point.

(d) *Aniline Point.*—The aniline point of wax may be readily determined, and individual readings do not normally differ by more than 0·2° C. It has been shown by Lord [10] that there exists between the melting point and aniline point of commercial waxes a relationship which can be expressed by the equation

$$\text{Aniline Point (° C.)} = 0{\cdot}319 \times \text{Melting Point (° F.)} + 75{\cdot}6.$$

Thus, an increase of 0·319° C. in aniline point represents an increase of 1° F. in melting point, and assuming that aniline point may be determined

to ± 0·1° C., the possible experimental error would then be equivalent to ± 0·3° F. in melting point.

Aniline point is therefore the most promising of the properties so far reviewed, and is likely to lead to more accurate results than any other of these properties. The linear relationship between aniline point and melting point is, however, of special significance, as this means that if melting point does not exactly follow the mixture rule, then aniline point also behaves in the same way. Now a modification of the I.P.T. Setting Point Method (Serial Designation P.S. 11a) has been developed which may be used to determine melting point with an accuracy of ± 0·05° F., and it is clear, therefore, that no advantage is to be gained by using the property of aniline point in preference to melting point, especially as the latter is quite as easy to determine.

(e) *Melting Point.*—It was found that the melting point of a mixture of waxes could not be calculated accurately from the rule of mixtures. Blends of various waxes were found to have melting points slightly higher than the values calculated from the rule of mixtures. This was confirmed by the determination of the melting points (using I.P.T. Setting Point Method) for blends of two refined commercial waxes *H* and *L* of melting points 130·95° F. and 108·35° F., respectively, over the complete range 100 per cent. *H* to 100 per cent. *L*. The actual melting point and that calculated from the rule of mixtures are given for each of the blends in Table IV below and are represented graphically in Fig. 3.

TABLE IV.

% *H*.	M.Pt. ° F. (actual).	M.Pt. ° F. (calculated).	Difference between actual and calculated.
100	130·95	—	—
90	129·15	128·7	+ 0·45
80	126·95	126·4	+ 0·55
70	124·9	124·2	+ 0·7
50	120·4	119·65	+ 0·75
40	118·0	117·4	+ 0·6
30	115·65	115·1	+ 0·45
15	112·1	111·75	+ 0·35

This is further illustrated by the following results given by Berne-Allen and Work for certain wax blends. The values calculated by the law of mixtures are included.

TABLE V.

Blend.	Actual M.Pt. (Berne-Allen).	Calculated.
Equal wts. of 52·8° C. and 64·4° C. M.Pt. waxes .	59·1° C.	58·6° C.
Equal wts. of waxes of following M.Pts. 49·9, 52·8, 55·6, 60·3 and 64·4° C.	56·9° C.	56·6° C.

It will be noted that the experimental is slightly higher than the calculated value.

As would be expected, it is found that the smaller the difference between the melting points of the two stock waxes, the smaller is the deviation

between the actual and calculated values. For example, a 50/50 blend of two commercial waxes of melting points 130·95° F. and 119·85° F., respectively, was found to have a melting point of 125·6° F., compared with the calculated figure of 125·4° F.—a difference of 0·2° F.—whereas the difference between actual and calculated values for a 50/50 blend of 130·95° F. and 108·35° F. waxes is 0·75° F. (Table IV).

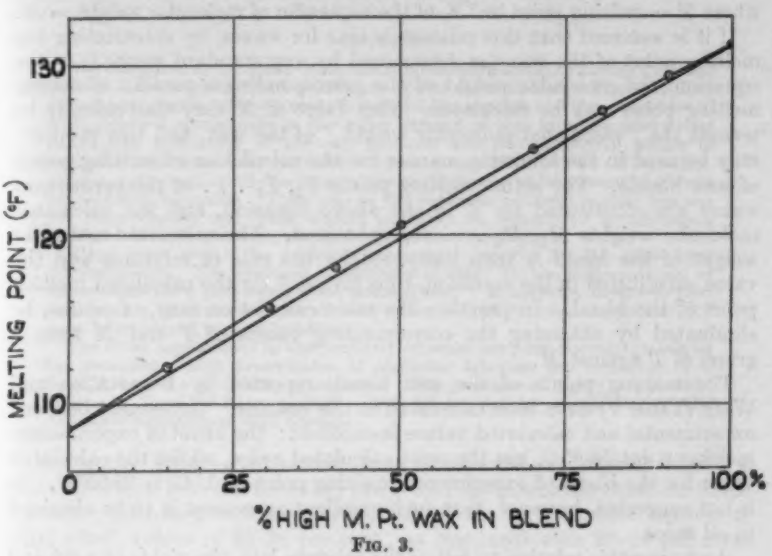

FIG. 3.

It is to be concluded that melting point is the most suitable physical property for the characterization of waxes, since :—

(*a*) it may be determined without difficulty to within $\pm$ 0·05° F., using a simple apparatus, and

(*b*) although not strictly following the mixture law, there is not a great difference between the experimental values and those calculated on the assumption that it does.

There is a further justification for its use, for although refined paraffin wax must meet definite minimum oil content and colour requirements, it is marketed primarily on the basis of melting point specifications.

MELTING POINT OF WAX AS A FUNCTION OF MOLECULAR WEIGHT.

Molecular weight strictly follows the mixture rule, and although, as already stated, its direct use is not considered suitable in this work, owing to the comparatively large experimental errors involved in its determination, the possibility of its indirect use was considered.

Now, the melting point/composition curve for mixtures of two waxes is similar in form to the melting point/molecular weight curve for the *n*-

paraffins within the wax range. It has been shown in a previous publication [11] that the relationship between molecular weight and melting point for these *n*-paraffins may be expressed as follows :—

$$T = \frac{414{\cdot}5\,M}{M + 94{\cdot}4}$$

where T = melting point in ° K of the *n*-paraffin of molecular weight = M.

If it is assumed that this relation is true for waxes, by substituting the melting point of the wax (as determined by any standard method) in the equation, the molecular weight of the corresponding *n*-paraffin of similar melting point may be calculated. This value of M may conveniently be termed the " calculated molecular weight " of the wax, and this property may be used in the following manner for the calculation of melting points of wax blends. The actual melting points T_1, T_2 . . . of the constituent waxes are substituted for T in the above equation, and the calculated molecular weights M_1, M_2 . . . thus obtained. The calculated molecular weight of the blend is then computed by the rule of mixtures and the value substituted in the equation, thus giving T for the calculated melting point of the blend. In practice, the main calculation may, of course, be eliminated by obtaining the corresponding values of T and M from a graph of T against M.

The melting points of the wax blends reported by Berne-Allen and Work (Table V) have been calculated in this manner. Agreement between experimental and calculated values is excellent; the blend of experimental melting point 56·9° C. has the same calculated value, whilst the calculated value for the blend of experimental melting point 59·1° C. is 59·05° C. It is not suggested, however, that such excellent agreement is to be obtained in all cases.

Assuming this relation to follow the mixture law, the yields of solid and liquid phases obtained in many equilibrium experiments (described in Part II) were calculated, and it was found that the agreement between experimental and calculated figures was, in general, closer than when melting point alone was used. The use of this relation for the wax employed in this research is therefore justified.

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RESEARCHES ON ASPHALTENES. PART I.*

By F. J. Nellensteyn and J. P. Kuipers.

Summary.

The difference in solubility of asphaltic bitumen in solvents having the same surface tension (ether and petroleum spirit) is explained. The solubility curves of asphaltic bitumen from Trinidad-Lake asphalt and the separation of light-coloured compounds in the ether extract of this bitumen prove that incomplete miscibility of the oily medium and the protective bodies of asphaltic bitumens with petroleum spirit enters into action. The ether-soluble, petroleum-spirit-insoluble compounds appear to be present in almost all asphaltic bitumens. Their rôle in the asphaltic bitumen system has not yet been cleared up; they should probably be considered as belonging partly to the oily medium, partly to the protective bodies.

Ethyl ether should be preferred to normal gasoline for the asphaltene determination. The practical advantages of ether for the determination of asphaltenes have previously been pointed out.[1] It appears, however, that also from a theoretical point of view ether should be used instead of the usually employed normal gasoline.

The ether asphaltenes in the majority of cases are pure flocculates, whilst the petroleum-spirit precipitates of asphaltic bitumen are mixtures of a flocculate with a de-mixing precipitate. The presence of highly oxygenated bodies in Trinidad-Lake asphalt could be proved by extraction of the difference-asphaltenes with acetone.

Asphaltic bitumen extracted from Trinidad-Lake asphalt shows a great difference between the ethyl ether- and the petroleum ether-insoluble content. With a petroleum spirit of about the same surface tension as ethyl ether, values of 35–38 per cent., as compared with 20–24 per cent. for ethyl ether, are found. Although a quantitative relation between the surface tension of the flocculating agent and the amount of precipitate, as indicated by the surface-tension rule,[1] has been found[2] only for tar, generally also for asphaltic bitumen the lower surface-tension liquids cause a higher amount of precipitate. The great difference between the petroleum spirit and the ether precipitate therefore indicates an incomplete miscibility of the medium and the protective bodies of Trinidad asphaltic bitumen with petroleum spirit. In this case it is easy to prove that incomplete miscibility enters into action. The filtrate of the ether asphaltenes, after evaporation of the ether, gives with petroleum spirit a *rather light-coloured* precipitate.

Further, this incomplete miscibility of petroleum spirits may be deduced from the curves in Fig. 1. The precipitates are obtained in the following way :—

2 gm. of asphaltic bitumen are mixed in an Erlenmeyer with 100 mls. of the precipitating mixtures. The bitumen is divided into small pieces with a glass rod, and the Erlenmeyer left in a cool place ($\pm$ 5° C.) overnight. The precipitate is filtered through a weighed Schleicher and Schüll filter no. 589,[2] and washed with 200 mls. of the precipitating liquid. When

* Paper received March 1940.

high-boiling liquids are used (petroleum spirit 80–100, 100–130 and their mixtures) the precipitate should be finally washed with 15 mls. pentane. The filter is dried, weighed, and burnt. The amount of precipitate is calculated from the difference in weight of the filter and the weight of the ashes.

The precipitating mixtures are composed of ether, various kinds of petroleum spirit, and mixtures of these liquids. The gasolines used have the following properties :—

	Specific gravity at 25°.	Surface tension dyne/cm. at 25°.
Petroleum spirit 40–60° . .	0·6482	17·5
,, ,, 60–80° . .	0·6838	21·2
,, ,, 80–100° . .	0·7085	22·7
,, ,, 100–130° . .	0·7313	24·3

Table I shows the amount of precipitate which various mixtures give with asphaltic bitumen from Trinidad-Lake asphalt.

TABLE I.

Percentage of Precipitate.

Ethyl ether 20·0%.

Petroleum spirit, b.p.	Ethyl ether–petroleum spirit mixtures			100% petroleum spirit.
	3 : 1	1 : 1	1 : 3	
40–60°	22·3	24·2	27·8	38·0
60–80°	19·1	18·7	21·0	27·2
80–100°	17·5	17·4	19·9	25·9
100–130°	15·2	14·3	17·3	21·1

In previous publications,[3] attention was directed to the fact that the combination of a flocculent with a partly miscible liquid of high surface tension leads to a solubility in the mixtures which may be higher than the solubility in both components separately. Conversely, as in the case of the curves in Fig. 1, one may conclude that incomplete miscibility of one of the components with the bitumen occurs if the concentration–insolubility curve shows a minimum or a marked deviation from the straight line connecting the values for the pure liquids (*AB*, *AC*, etc.). Here petroleum spirit is the incompletely miscible component.

From the foregoing it clearly appears that the great difference in solubility of asphaltic bitumen extracted from Trinidad Lake asphalt in ethyl ether and petroleum spirit of the same surface tension is in complete concordance with the surface-tension rule, the higher value for petroleum spirit being due to incomplete miscibility.

Little is known about the nature and amount of these petroleum-spirit-insoluble, ethyl-ether-soluble compounds, owing to the fact that in the ordinary asphaltene determination petroleum spirit is used as flocculent, and a mixture of the ether asphaltenes with these compounds is always

obtained. Therefore it seemed important to determine the amount of the ether-soluble, petroleum-spirit-insoluble compounds in various asphaltic bitumens. The following method has been used :—

The ether asphaltenes are precipitated according to the methods described on pages 401–2. The filtrate, after evaporation of the ether on a steam bath, is mixed with 100 mls. petroleum spirit (boiling point 40–60° C.). After standing over-night in a cool place (± 5° C.), the precipitate is filtered through a weighed Schleicher and Schüll filter no. 589 [2] and washed with 200 mls. petroleum spirit. The filter is dried, weighed, and burnt. The amount of precipitate is calculated from the difference in weight of the filter and the weight of the ashes.

The result of these determinations is given in Table II.

TABLE II.

Asphaltic bitumen from	Method of manufacturing.	Softening point ring and ball, ° C.	Ether-asphaltenes, %.	Petroleum spirit 40–60 asphaltenes, %.	Petroleum spirit-ether-asphaltenes.	
					Calculated, %.	Determined, %.
Trinidad-Lake Asphalt.	Extracted with CS_2	65·0	21·0	38·0	17·0	11·0
Boeton asphalt						
(*a*) Kaboengka .	"	105·0	33·2	47·4	9·2	6·7
(*b*) Waritl . .	"	84·0	25·3	38·9	13·6	8·7
Gilsonite . . .	No treatment	—	41·6	62·2	20·6	7·2
Californian crude .	Steam refined	49·0	6·1	17·6	11·5	10·7
Rumanian crude .	" "	55·0	17·3	25·3	8·0	6·8
South Mexican crude .	" "	57·0	23·1	29·1	6·0	4·7
Egyptian crude . .	" "	53·1	22·6	34·6	12·0	8·0
Venezuelan crude .	" "	55·0	17·2	21·5	4·3	4·9
Dutch East-Indian crude	" "	48·0	12·2	20·7	8·5	6·1
Mexican crude . .	" "	118·0	39·3	47·0	7·7	6·1
" " . .	" "	63·5	25·2	29·2	4·0	5·4
" " . .	" "	56·0	22·4	27·8	5·4	4·1
" " . .	" "	53·6	21·8	25·6	3·8	5·2
" " . .	" "	47·6	20·8	24·8	4·0	6·3
Miri crude . .	" "	52·5	1·3	5·7	4·4	5·5
Mesopotamia . .	No treatment	52·5	21·2	29·6	8·4	5·7
Venezuelan crude .	Blown with air	57·8	21·6	25·4	3·8	0·9
Mexican crude . .	" "	64·0	29·5	37·4	7·9	3·4
" " . .	" "	175·0	48·2	55·6	7·4	1·5

It is evident that the presence in bitumens of compounds which are insoluble in petroleum spirit and soluble in ether is a generally occurring phenomenon certainly not restricted to the natural asphalts, although some of these—*e.g.*, Trinidad-Lake asphalt show a very high content of these bodies. Owing to this general occurrence it seems desirable to look for a name for these compounds. According to the Dutch nomenclature, all the flocculates and semi-flocculates of asphaltic bitumen are called asphaltenes; the name of the flocculent is to be added. So these ether-soluble, petroleum-spirit-insoluble compounds should be called (*petroleum-spirit* 40/60-*ether*) *asphaltenes* or, briefly, "*difference-asphaltenes*."

Table II demonstrates some striking facts. In the first place, the amount of difference-asphaltenes is much more constant than that of the ether- or petroleum spirit-asphaltenes. No correlation between the ether asphaltenes content and the amount of difference-asphaltenes exists. Asphaltic bitumens from the same crude (Mexican) with increasing softening

point show about the same amount of difference-asphaltenes, whilst the ether asphaltenes increase from 20·8 to 39·3 per cent. Although these determinations are satisfactorily reproducible, it must be taken into consideration that the amount of precipitate depends greatly on the mode of operation.

These are conventional determinations; therefore a warning should be issued against going too far in reaching conclusions. The accuracy of the difference-asphaltenes determination is about $\pm$ 1 per cent., so that values of 4–6 per cent. should be considered as practically constant. The most important fact is the general occurrence of these difference-asphaltenes in asphaltic bitumens; the highest values are found for Trinidad-Lake asphalt

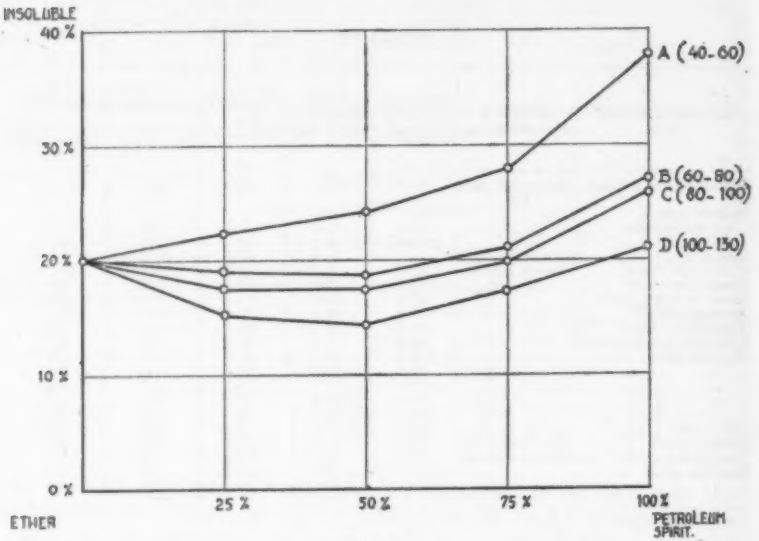

FIG. 1.

and Californian asphaltic bitumen, the lower values for the blown products. Miri asphaltic bitumen contains a normal percentage of difference-asphaltenes, but its ether asphaltene-content is low.

The calculated and determined values of the difference-asphaltenes are not the same; in most cases the determined values are lower. This may be caused by adsorption of the difference-asphaltenes by the micelle-kernels.

Generally the amount of ether asphaltenes is smaller than that of the flocculates of petroleum spirit with about the same surface tension. It is, however, possible, in connection with the fact that the nature of the compounds in asphaltic bitumen may vary considerably, that the ether-asphaltenes content will be found higher than that of the petroleum-spirit asphaltenes. We know only one such exceptional case; an asphaltic

bitumen from an Argentine crude with 17·2 per cent. ether- and 13·3 per cent. petroleum-spirit asphaltenes.

The rôle of the difference-asphaltenes is not yet cleared up; as a demixing product they may belong to the oily medium, but, on the other hand, they show peptizing properties for fine mineral powders. An analysis of a highly altered asphaltic bitumen (from the excavations at Mohenjo-Daro,[4] Indus valley, 3000 B.C.) gave a similar indication. This 5000-years-old product had become partly insoluble in CS_2; the soluble part showed the following asphaltene content :—

	Per cent.
Ether asphaltenes	42·0
Petroleum spirit (40–60° C.) asphaltenes	67·7
Difference-asphaltenes	26·0

The high amount of difference-asphaltenes is an indication of the resistance of these bodies against alteration, but also of their peptizing properties; even the highly altered micelles may be peptized by the difference-asphaltenes. As peptizing compounds they belong to the protective bodies, and so to the micelle; but the fact that the difference-asphaltenes are easily separated from the micelles by ether proves that they belong at least partly to the oily medium.

The difference-asphaltenes from Trinidad Lake asphalt may be separated into an acetone-soluble part, a dark red amorphous body, which dissolves into acetone with a cherry-red colour, and a brown residue. On adding petroleum-spirit, the colour of the acetone solution changes into light brown; with more petroleum spirit a precipitate is obtained, the colour of which is lighter than that of the original product. An attempt to purify the acetone-soluble bodies by mixing with petroleum spirit to which some acetone had been added gave no result.

A lighter-coloured product could be obtained by distilling Trinidad-Lake asphalt in high vacuum and precipitating the distillate with petroleum spirit 40–60° C. The precipitate is washed with petroleum spirit; it dissolves in ether.

A comparison of the elementary composition of the ether asphaltenes, the difference-asphaltenes, the acetone-soluble compounds from the difference-asphaltenes and the petroleum-spirit insoluble compounds from Trinidad-Lake asphalt gives interesting indications regarding the nature of the difference-asphaltenes.

The ether asphaltenes obtained in the ordinary way from Trinidad-Lake asphalt still contain a considerable amount of ashes and ether. The product is purified in the following way :—

20 gm. of crude ether asphaltenes are dissolved in 1 : 1 benzene; after standing for a week, the liquid is decanted as far as possible and filtered through a fine filter. The filtrate is concentrated to a quarter of the volume and precipitated with ether. The precipitate is filtered and washed with ether until a colourless filtrate is obtained. The ether asphaltenes are dried at 100–105° C. and brought into a U-tube, heated in a water-bath at 60° C. A stream of dry pure nitrogen is led through the asphaltenes, by which the last traces of ether and water are eliminated.

Elementary Composition of Products from Trinidad-Lake Asphalt.

	C.	H.	S.	N.	Ashes.	O (by difference).
1. Ether asphaltenes . .	78·1	7·6	7·4	1·6	0·9	4·4
2. Difference-asphaltenes .	78·8	8·3	5·8	1·3	0·0	5·8
3. Petroleum-spirit insoluble from high vacuum distillate . . .	74·8	6·7	4·3	2·6	0·7	10·9
4. Acetone-soluble part of the difference-asphaltenes .	74·8	7·7	6·2	1·5	0·0	9·8

The most striking fact is the high oxygen content of the acetone-soluble part of the difference-asphaltenes. These highly oxygenated bodies are also to be found in the petroleum-spirit-insoluble part of a Trinidad-Lake asphalt high-vacuum distillate. It appears that in Trinidad-Lake asphalt the O-compounds are concentrated in the difference-asphaltenes, the S-compounds in the ether asphaltenes.

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EVALUATION OF ROTARY DRILLING LINES.*

By H. C. H. Darley, B.Sc., A.M.Inst.Pet.

Introduction.

The main reasons for keeping records of the ton-miles done by drilling lines are :

(1) To obtain a comparison of the service given by different brands of lines.

(2) As a guide to the best treatment to give lines.

(3) As an indication of how much more life may be expected from a line.

The chief objection is that it is not practical to determine the work done by certain forces such as the dynamic forces involved, or as the work done when pulling on stuck pipe. If it is considered worth while keeping records, there remains the question of what limit of accuracy is justified in view of these indeterminate stresses. In the author's opinion the determinations should be as accurate as possible, as long as they can be made quickly each day from simple charts.

A paper on the " Evaluation of Rotary-Drilling Lines " was published by K. N. Saatjian and V. V. Mason in the *Oil Weekly* of 18th November, 1935. They surveyed the effect of the various factors and equipment throughout the U.S.A., and produced an alignment chart based on a modified form of Hallam Anderson's formula. This chart has been approved by the A.P.I.

It will be seen at once that, since this chart is based on data which represent the average of all the fields in the U.S.A., it cannot be expected to give accurate results when applied to a particular field. The author therefore prepared graphs based on a modified formula to suit the conditions on the Apex field.

The Formula.

The formula on which these graphs were based is as follows :

Work Done by Drill Pipe.

Let n = the number of stands.
l = length of one stand in feet.
L = length of string in feet.
w = weight of pipe in lb./ft. (with weight of tool joints prorated over the whole).

Running in, work done by first stand $= wl \times L$
" " " " " 2nd " $= wl \times (L - 1)$
" " " " " nth " $= wl \times \{L - (n - 1)1\}$

* Paper read to a Meeting of the Trinidad Branch on 28th February, 1940.

By arithmetic progression, work done by whole string:

$$= \frac{n}{2}[wlL + wl\{L - (n-1)l\}]$$

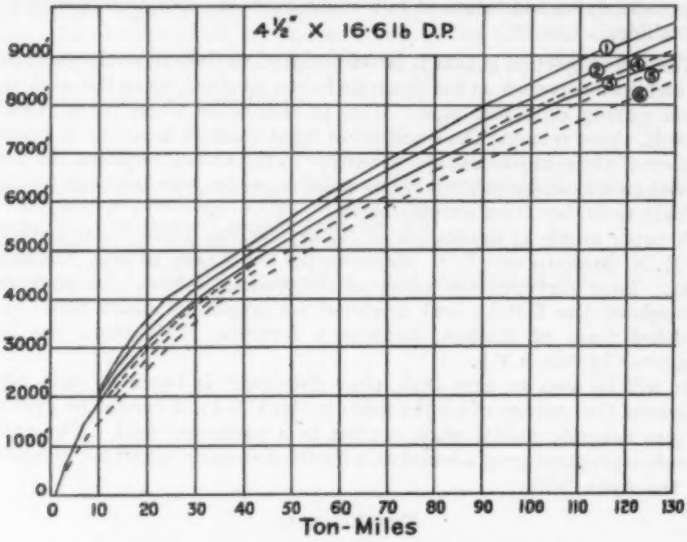
$$= \frac{n}{2} wl\{2L - (n-1)l\}$$

$$= nwlL - \frac{n^2}{2} wl^2 + \frac{n}{2} wl^2$$

$$= wl^2 - \frac{wL^2}{2} + \frac{wLl}{2}$$

$$= \frac{wL}{2}(L+1)$$

APEX (TRINIDAD) OILFIELDS, LTD., TON MILE CHART.



Curve 1,	Block and hook	6,000	lb.	Mud	125	lb./cu. ft.
Curve 2,	,,	,,	6,000	,,	,,	100	,, ,,
Curve 3,	,,	,,	6,000	,,	,,	75	,, ,,
Curve 4,	,,	,,	10,000	,,	,,	125	,, ,,
Curve 5,	,,	,,	10,000	,,	,,	100	,, ,,
Curve 6,	,,	,,	10,000	,,	,,	75	,, ,,

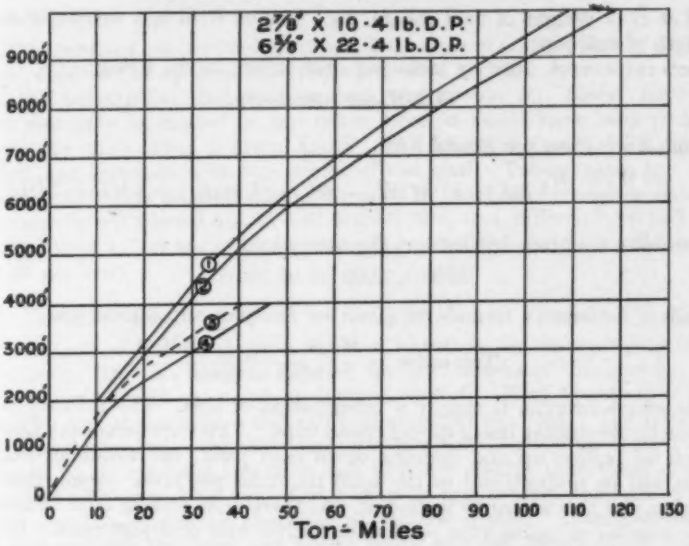
Hence work done by drill pipe for one round trip $= wL(L+1)$.

The weight per foot must be corrected for the buoyancy of the steel in the mud. The correction is given by the volume of the steel per foot multiplied by the specific gravity of the mud. Saatjian and Mason subtracted only half this correction in their calculations. The author does not see the reason for this, and cannot agree with it. In support of this it may be stated that their tables, derived by this means, giving the weights

of drill pipe in various weights of mud, do not agree with those published in the *Petroleum Engineer's Continuous Tables* (July 1939 and subsequent issues). If the pipe is floated in, the effective weight per foot will be less again, the correction being, of course, the overall volume per foot multiplied by the specific gravity of the mud. If the pipe is floated in, but filled to the surface after a certain length has been run the formula must be altered as follows :

Let W_1 = Weight/ft. of drill pipe in mud fluid full.
W_0 = „ „ „ „ „ „ „ „ empty.
L_1 = Depth when pipe is first filled.
L_2 = „ „ „ „ filled for second time.

APEX (TRINIDAD) OILFIELDS, LTD., TON MILE CHART.



Curve 1, 2⅞" D.P. Block and hook 10,000 lb. Mud 125 lb./cu. ft.
Curve 2, 2½" „ „ „ 10,000 „ „ 100 „ „
Curve 3, 6⅝" „ „ „ 6,000 „ „ 75 „ „
Curve 4, 6⅝" „ „ „ 10,000 „ „ 75 „ „

Suppose the pipe were run in all the way empty, then the work done :

$$= \tfrac{1}{2}W_0L(L+1)$$

Correction for portion filled :

$$= (W_1 - W_0)L_1(L - L_1)$$

Further correction if pipe is filled a second time :

$$= (W_1 - W)L_1(L - L_2)$$

Work done pulling out :

$$= \tfrac{1}{2}W_1L(L+1) \text{ as before.}$$

Hence for round trip:

$$\frac{W_1 + W_0}{2} L(L+1) + (W_1 - W_0)L_1(L - L_1) + (W_1 - W_0)L_1(L - L_2).$$

Work Done by Travelling Block.

For every stand block travels 21 feet
" " round trip block travels $2 - 2nl$ feet
$= 4L$
And work done $= 4ML$
When M = weight of block, hook, etc.

Work Done by Drill Collars, Reamers, etc.

Let D = Weight of drill collars, etc., in mud fluid less weight of same length of drill pipe.
Then extra work done by including drill collars in the string:
$= 2DL$ per round trip.

Total Work Done per Round Trip.

$$= \frac{W_1 + W_0}{2} L(L+1) + (W_1 - W_0)L_1(L - L_1) + 4ML + 2DL.$$

Ton-miles are given by dividing the above by:
5280×2000 (short tons).

Hallam Anderson's formula as given by Saatjian and Mason was:

$$\text{Ton-miles} = \frac{WL(1+L) + 4LM}{5280 \times 2000}$$

The above formula is simply a modification of this. The primary work done by the drilling line is during round trips. There are secondary factors, such as pulling up and reaming down each joint, but ton-miles due to this will be proportional to the total ton-miles per hole. Again there is casing, but this is largely floated in, and there is no pulling out. Further, the number of casing runs are few compared with drill-pipe runs. It can therefore be seen that the number of ton-miles due to running casing will form only a small fraction of the total ton-miles. It is therefore held that by recording the ton-miles done by each round trip by means of the above formula will give a figure closely proportional to the actual ton-miles done by a wire line within the limits set at the beginning of this paper.

The Construction of Graphs.

For quick daily determinations a series of graphs were prepared. The many variable factors necessitate a number of curves, but for easy reading this number must be kept low. The effect of these variables was therefore investigated, and the number of curves selected for each variable was according to the magnitude of its effect. One curve was then drawn to represent the average of each set of conditions normally liable to be encountered on the Apex Field. The effect of these variables is as follows:

Weight of Mud.

Either a shale mud weighing between 72 and 78 lb./cu. ft. or a loaded mud between 90 and 110 lb./cu. ft. is normally used in this field. An average curve is therefore provided for each of these groups and one to cover the occasional use of heavy mud. The main effect of the weight of the mud is due to floating in, *e.g.*, suppose an average curve of 100 lb./cu. ft. was used instead of the 3 curves. With 4½-inch drill pipe at 4000 feet, a light block and 75 lb. mud, the error would be 12 per cent. if the pipe was floated in. If the pipe was not floated, the error would only be 4 per cent. Saatjian and Mason did not allow for floating in. Also they considered that effect of tool joints and buoyancy of the metal counteracted each other, and so allowed for neither.

Floating in.

The standard practice here is not to float more than 6000 feet of 2⅞-inch pipe, 4000 feet of 4½-inch, 2000 feet of 6⅝-inch, and the curves were drawn on the assumption that this amount was floated in. Since, however, less pipe may be floated in, but never more, it would have been perhaps better to have taken a lower figure. The effect of floating the pipe is large, and increases with the weight of the mud. Taking again the case 4½-inch drill pipe at 4000 feet, with a 75-lb. mud, the ton-miles done if the pipe is not floated are 34·5, if floated 29·8, or a difference of 15·7 per cent. With 125 lb. mud the respective figures are 31·9 and 24·0, a difference of 33 per cent.

The Travelling Block.

Saatjian and Mason suggest a figure of 10,000 lb. for the travelling block and hook. On this field the heavier rigs have blocks of this size but the lighter rigs have blocks weighing down to 6000 lb. With 4½-inch drill pipe at 2000 feet this means a difference of some 30 per cent. The effect of the travelling block at shallow depths is therefore large (with 4½-inch drill pipe in 75 lb. mud the ton-miles due to it are greater than those due to the drill pipe up to 3000 feet), and it must be remembered that a wire line may spend its entire life operating at shallow depths. Therefore curves were drawn for the light and heavy hooks and any intermediate values interpolated.

Drill Collars.

1500 lb. extra are allowed for drill collars with 2⅞-inch drill pipe, 2500 lb. for 4½-inch drill pipe and 5500 lb. with 6½-inch drill pipe. These weights allow for the buoyancy of the mud. At the most (say 6⅝-inch drill pipe at 2000 feet) these weights represent only 13 per cent. of the ton-miles. Hence a 50 per cent. variation in the weight of the drill collars will give only a 6 per cent. variation in ton-miles at maximum. Hence no variation in size and length of drill-collar sections was allowed for. Saatjian and Mason allow for very much heavier drill collars than these. They made a flat addition of 12 ton-miles per round trip in all determinations. This would lead to serious errors in this field, especially at shallow depths, since the total ton-miles up to 2000 feet is usually less than this.

Conclusions.

Readings made from the alignment chart of Saatjian and Mason are, on a broad average, 50 per cent. higher than those obtained from the author's curves. This is quite understandable, since, as the above discussion shows, in every point of difference their methods give the higher readings.

It may be argued that one average curve for all the conditions on this field would be enough. The objection to this is that a wire line is assigned to a particular rig, and that rig often to a particular type of work, and so the line would operate all its life under one particular set of conditions. Therefore results from the single average curve would be accurate only over the average of a number of lines. A curve for each set of conditions gives a reasonably accurate figure for each line and causes little extra trouble in reading. Since the effect of the dynamic stresses must mainly depend on weight, their effect would be proportional to the static figure, and hence results should still be comparative. This, of course, presupposes that the brake is handled reasonably. If not, the line will wear much more rapidly, since impulse loads are very much higher than static. For unusual strains, such as pulling casing, it is suggested that a note be made against any line undergoing same.

Finally the author again emphasizes that the degree of accuracy which is justified is an open question. This paper submits one view, and it is hoped that others will express theirs. If the value of fairly accurate ton-mile records is admitted, it might be worth investigating the best method of making them comparative on an Island basis.

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